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TOTAL HEMISPHERICAL EMITTANCE MEASUREMENTS
OVER THE TEMPERATURE RANGE 77°K TO 300°K

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ADMINISTRATIVE INFORMATION

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ABSTRACT

Total hemispherical emittance measurements were made on a series of high emittance black paints, metal blacks and anodized aluminum over the temperature range from 77°K to 300°K by measuring the rate of temperature decay of a coated disk in a vacuum. Analytical and experimental analysis show that errors inherent in the method are less than 1% of the emittance of a black body at the temperature of the measurement, although instrumental errors and uncertainties in the heat capacities of the specimens reduce the accuracy of the reported data to $\pm 10\%$. The blackest material measured was electrically deposited platinum black on gold. The emittance of a black paint appeared to be more dependent on the thickness that it could be applied than on the specific type of paint. Heavily anodized aluminum exhibited an emittance near 77°K of about 0.6, nearly as high as the best black paints, although it looked bright to the eye. The best platinum black coating had an emittance above 0.9 at 77°K. The metal blacks and the black paints had emittances above 0.95 at room temperature.

This technical documentary report has been reviewed and is approved.

~~FOR THE COMMANDER~~

for John C. Fischer, Major USAF
PAUL L. FISHBURNE
Colonel, USAF
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INTRODUCTION

The use of passive cooling by radiation of infra-red detectors in space requires detailed data on the total hemispherical emittance of the detector and its surrounding components. To minimize reflection, to increase the heat losses from the system to the maximum and to produce the lowest possible detector temperature, the system should be "black" at the operating temperature. Black means behaving as a perfect Planckian radiator. Existing theory is virtually powerless to predict the radiation properties of high emittance materials at temperatures near 100°K from data taken at 300°K and above. Consequently, an experimental program to directly measure these properties for certain apparently useful materials was undertaken at the Naval Radiological Defense Laboratory.

The experimental measurements of this program were obtained by suspending appropriate specimens in an evacuated chamber with walls at liquid nitrogen (L/N) or liquid helium (L/He) temperature and measuring the rate of temperature decay. If the chamber is properly designed and operated, the emittance ϵ of the specimen is given by

$$\epsilon = \frac{m C \dot{T}}{A_1 \sigma T_s^4}$$

where m is the mass of the specimen; C , the heat capacity at constant pressure and the temperature of the specimen; \dot{T} , the rate of temperature decay with respect to time; A_1 , the radiating area of the specimen; σ , the Stefan-Boltzmann constant; and T_s , the specimen temperature. The

criteria for the design and operation of such a chamber are discussed in the next sections of this report.

THEORY OF EXPERIMENT

The total rate of heat loss, Q_t , from a specimen suspended in a chamber with walls at a temperature, T_w , lower than the specimen temperature, T_s , can be represented by

$$Q_t = Q_r + Q_s + Q_a + Q_c - Q_w \quad (1)$$

where Q_r , Q_s , Q_a and Q_c represent that portion of the heat lost by radiation, support conduction, air conduction and air convection, respectively, and Q_w represents the heat absorbed from the walls.

For vacuums of 10^{-4} Torr or less, where the mean free path of the air molecules exceed the dimensions of the chamber, air currents cannot exist and Q_c is zero. Free molecular conduction losses can be approximated from a formula given by Dushman (1) for coaxial cylinders.

$$Q_a = J B P \left(\frac{272.2}{T_w} \right)^{1/2} (T_s - T_w) \quad (2)$$

where J is a constant related to the surfaces and is near 1, B is the molecular heat conductivity and is about 12×10^{-6} watt cm^{-2} $^{\circ}\text{K}^{-1}$ μb^{-1} for air, P is the pressure in microbars ($1\mu\text{B} \approx 0.75 \times 10^{-3}$ Torr) and Q_a is the energy lost from the specimen in watts cm^{-2} . This equation is probably not completely valid for the experimental apparatus used, but the estimate of heat losses by air conduction are relatively independent of chamber size and shape as long as the mean free path of the air

molecules exceeds the dimensions of the chamber. Substituting numbers in equation (2) indicates that a pressure of not more than 3×10^{-8} Torr must be achieved if the heat losses due to air conduction are to be held to less than 1% of the radiation losses from a black specimen at 100°K.

Conductive losses down the specimen supports may be written as

$$Q_s = K \Delta T S/L \quad (3)$$

where K is the effective thermal conductivity of the supports, S is their cross sectional area, L is their length and ΔT is the temperature difference over the length, L. The thermal conductivity, K, is a generally increasing function of temperature for alloys below 300°K and, for most alloys, the thermal conductivity at 4°K is less than 10% of its room temperature value. Because of the strong temperature dependence of K and the complete lack of published experimental data on K for thermocouple materials (except the pure metals) Q_s can only be evaluated for a specific apparatus by experiment. Once Q_s has been determined, however, variations in the length or size of the supports can be compensated for by linear corrections in S and L.

While it is apparent that the correction in Q_s should be linear with respect to the support wire area, the linearity with respect to the reciprocal of the wire length is not as obvious. Such linearity can be shown as follows:

$$Q_s = K(T)S \frac{dT}{dx} \quad (4)$$

where $K(T)$ represents the thermal conductivity as a function of temperature and x is the distance along the wire.

Then

$$S \int_{T_s}^{T_w} K(T) dT = Q_s \int_0^L dx$$

$$S \int_{T_s}^{T_w} K(T) dT = Q_s L$$

and

$$Q_s = \frac{S}{L} \int_{T_s}^{T_w} K(T) dT = \bar{K} \Delta T \frac{S}{L}$$

where \bar{K} is the average value of the thermal conductivity over the temperature range T_w to T_s , so that $Q_s \sim \frac{S}{L}$ for a given type of support.

The heat loss due to radiation is,

$$Q_r = A_1 \epsilon \sigma T_s^4 \quad (5)$$

The design of the experimental apparatus is to make Q_r the only significant heat loss.

Heat absorbed from the wall Q_w is made up of two parts: that radiated directly from the wall and absorbed by the specimen Q_{ew} and that radiated by the specimen and reflected from the wall Q_{rw} .

$$Q_w = Q_{ew} + Q_{rw} \quad (6)$$

$$Q_{ew} = A_1 \epsilon_c \sigma T_w^4 \quad (7)$$

where $\bar{\epsilon}$ is the absorptance of the specimen to the wall radiation and ϵ_c is the effective emittance of the chamber as seen by the specimen.

An exact analysis of the effect of internal reflections in chambers as complex as those in the experimental apparatus would be extremely difficult, but with the aid of some simplifying assumptions a reasonable approximation can be made. Consider, therefore, a chamber with perfectly diffuse walls uniformly irradiated by direct radiation from the specimen. The optical properties of the walls do not vary with wavelength and the walls do not radiate significantly.

The flux leaving a differential area da_1 and falling on a differential area da_2 is given by

$$d\bar{\epsilon} = G (da_1 \cos \alpha_1) \frac{da_2 \cos \alpha_2}{D^2} \quad (8)$$

where G is the flux per unit area per unit solid angle in the direction normal to da_1 , α_1 is the angle between the normal to da_1 and a line connecting da_1 to da_2 , α_2 is the angle between the normal to da_2 and a line connecting da_2 and da_1 , and D is the distance between da_1 and da_2 . Throughout this discussion the subscript 1 refers to the specimen and the subscript 2 to the wall.

Equation (8) is based on a cosine distribution of the flux leaving the surfaces. The transfer between two finite areas A_1 and A_2 is given by

$$\bar{\epsilon}_{1,2} = \int_{A_1} \int_{A_2} \frac{G}{D^2} \cos \alpha_1 \cos \alpha_2 da_1 da_2 \quad (9)$$

The total flux leaving A_1 is equal to

$$\bar{\epsilon}_1 = \int_{A_1} \int_0^{\pi/2} G da_1 \cos \alpha_1 2\pi \sin \alpha_1 d\alpha_1 \quad (10)$$

$\sin \alpha_1 d\alpha_1$ may be written as $-d \cos \alpha_1$ and (10) becomes

$$\begin{aligned}\Phi_1 &= \int_{A_1} \int_0^{\pi/2} -2\pi G da_1 \cos \alpha_1 d \cos \alpha_1 \\ &= \int_{A_1} \pi G da_1 = \pi A_1 G\end{aligned}\quad (11)$$

The shape modules $F(1,2)$ of a system is defined as the fraction of the radiation which leaves surface 1 and falls directly on surface 2 without intermediate wall reflection.

$$\begin{aligned}F(1,2) &= \frac{\Phi_{1,2}}{\Phi_1} = \frac{1}{\pi A_1 G} \int_{A_1} \int_{A_2} \frac{G}{D^2} \cos \alpha_1 \cos \alpha_2 da_1 da_2 \\ &= \frac{1}{\pi A_1} \int_{A_1} \int_{A_2} \frac{\cos \alpha_1 \cos \alpha_2 da_1 da_2}{D^2}\end{aligned}\quad (12)$$

Since the system from A_2 to A_1 is symmetrical with A_1 to A_2 , then, by the same reasoning

$$F(2,1) = \frac{1}{\pi A_2} \int_{A_1} \int_{A_2} \frac{\cos \alpha_1 \cos \alpha_2 da_1 da_2}{D^2}\quad (13)$$

and therefore

$$A_1 F(1,2) = A_2 F(2,1)\quad (14)$$

This reciprocity relationship is needed for an analysis of the emittance chamber. In the emittance chamber, we consider two areas, that of the specimen A_1 and of the walls A_2 . Then $F(1,2) = 1$, since all the radiation leaving A_1 must strike the walls. Then from (14)

$$F(2,1) = \frac{A_1}{A_2}\quad (15)$$

and the shape modulus for direct transfer of energy from the wall to the

wall, $F(2,2)$ is

$$F(2,2) = 1 - \frac{A_1}{A_2} \quad (16)$$

The irradiance of the walls, E_2 , which includes all the energy striking the walls, is given by

$$A_2 E_2 = A_1 \epsilon_1 \sigma T_s^4 + (1 - \epsilon_1) E_1 A_1 + F(2,2) (1 - \epsilon_2) E_2 A_2 \quad (17)$$

where E_1 is the irradiation of the specimen by the walls. The first term on the right hand side in Eq. (17) represents the direct radiation from the specimen, the second term the reflected radiation from the specimen and the third term reflected radiation from the wall that misses the specimen. Similarly, E_1 is given by

$$E_1 A_1 = F(2,1)(1 - \epsilon_2) (E_2 A_2) \quad (18)$$

From (15), (16), (17), and (18)

$$\begin{aligned} E_1 A_1 &= \frac{A_1}{A_2} (1 - \epsilon_2) \left[A_1 \epsilon_1 \sigma T_s^4 + (1 - \epsilon_1) E_1 A_1 + \left(1 - \frac{A_1}{A_2}\right) (1 - \epsilon_2) E_2 A_2 \right] \\ E_1 &= (1 - \epsilon_2) \left[\frac{A_1}{A_2} \epsilon_1 \sigma T_s^4 + \frac{A_1}{A_2} (1 - \epsilon_1) E_1 + \left(1 - \frac{A_1}{A_2}\right) (1 - \epsilon_2) E_2 \right] \quad (19) \end{aligned}$$

From (15) and (18), $E_1 = (1 - \epsilon_2) E_2$ and from (16) and (19)

$$E_1 = (1 - \epsilon_2) \left[\frac{A_1}{A_2} \epsilon_1 \sigma T_s^4 + \frac{A_1}{A_2} (1 - \epsilon_1) E_1 + \left(1 - \frac{A_1}{A_2}\right) E_1 \right] \quad (20)$$

so

$$E_1 = \frac{\epsilon_1 \sigma T_s^4}{\frac{A_2}{A_1} \left(\frac{\epsilon_2}{1 - \epsilon_2} \right) + \epsilon_1} \quad (21)$$

Energy is reabsorbed at the rate

$$Q_{rw} = \frac{A_1 \epsilon_1^2 \sigma T_s^4}{\frac{A_2}{A_1} \left(\frac{\epsilon_2}{1 - \epsilon_2} \right) + \epsilon_1} \quad (22)$$

Equation (1) can now be rewritten, within the accuracy of the various approximations used to evaluate the various heat losses, as

$$Q_t = A_1 \epsilon_1 \sigma T_s^4 + \bar{K}(T_s - T_w)S/L + JBP(273.3/T_w)(T_s - T_w) - A_1 \epsilon_c \sigma T_w^4 - \frac{A_1 \epsilon_1^2 \sigma T_s^4}{\frac{A_2}{A_1} \left(\frac{\epsilon_2}{1 - \epsilon_2} \right) + \epsilon_1} \quad (23)$$

To determine ϵ_1 from Eq. (23) it is necessary that all terms except the first one be negligible, or, failing that, that they be accurately measured from the experiments. An attempt to evaluate how closely this goal is reached requires first a description of the experimental apparatus.

EXPERIMENTAL APPARATUS

The experiment consists of suspending a specimen in an evacuated black chamber with cold walls and measuring the rate of temperature decay. If the vacuum is good enough, the conductance of the suspending leads low enough, and if the chamber is actually a black body at a negligibly low temperature from the view point of the specimen, then the only way for the specimen to lose heat is by radiation, and the emittance of the specimen coating can be determined from its rate of temperature decay.

The apparatus was designed to cover emittance measurements over the range 77°K to 300°K. For the energy radiated from the walls to

be negligibly small compared to that radiated by the specimen at 77°K , the wall temperature had to be less than $1/3$ the specimen temperature. The simplest and safest way to obtain such a temperature is to use liquid helium (L/He) at 4°K and, consequently, both experimental chambers were designed in L/He dewars.

Figures 1 and 2 are semi-schematic drawings of Dewar I and its chamber. This dewar was on hand at the start of the project and was adapted to fit the experimental conditions. The working space inside the chamber was rather small and it was necessary to be extremely careful in the interior design, so that the chamber would be black at the wave lengths of interest. There were no parallel surfaces in the entire chamber, and the button was suspended at an angle and off center as shown. The dewar itself was of conventional design and would hold about five liters of L/He. Another five liters were required to cool the helium spaces after the entire dewar had been precooled to liquid nitrogen (L/N) temperature. This took from 12-15 liters of L/N.

Figures 3 and 4 show Dewar II, designed for the project and the arrangement of the working chamber. It was capable of testing two samples at one time. Unlike Dewar I, it had no electrical vacuum feed throughs at low temperature. The working volume of Dewar II was some 40-50 times that of Dewar I. This dewar was completed too late to contribute to the data in this report.

The specimens were suspended from one mil (.001") diameter chromel-alumel thermocouple wires. These terminated at insulated connections inside the chamber and were continued to the outside on wires of the same material but of heavier gage. Because the specimens swing very freely when so suspended, it was necessary to avoid excessive vibration or jarring of the dewar when data was being taken.

It took approximately 45 minutes to fill Dewar I with L/He. The dewar had to be precooled before adding the L/He to reduce the consumption of L/He and also to calibrate the specimen thermocouples, which required that the specimen be at L/N temperature. To reheat the specimen to near 300°K , a heater was provided in the chamber, positioned so as to fill as little as possible of the specimen field of view. This heater was a coil of five mil (.005") tungsten wire and it was heated to incandescence when used. The supports were blackened in the same manner as the rest of the chamber but the heater wire was bright, since all coatings burned off. The wire was round and therefore presented very little surface parallel to the specimen, and it was so small that it did not appreciably affect the measurements. The reference junction for the thermocouple was in L/N outside the L/He dewar. A L/N reference temperature reduced the effect of a reference temperature drift, since the emf of a chromel-alumel couple at 77°K is only $16\text{ }\mu\text{V}/^{\circ}\text{K}$ compared to $40\text{ }\mu\text{V}/^{\circ}\text{K}$ at 273°K . At the lower temperatures the thermocouple output was near zero with a L/N reference, consequently, it was possible to use the more sensitive ranges of the recording voltmeters. Boiling L/N does not exhibit the density gradients that exist in an ice bath and was probably more stable than an ice bath as a reference.

A dewar of L/N can be contaminated with oxygen from the air, which would change its boiling point, so care had to be taken to exclude air from the surface of the L/N, and to replace the used L/N at least daily. Additional L/N was never added to the reference dewar but the contents of the dewar were discarded and new L/N used.

The output of the thermocouple was connected to either a Dymec 2401A digital voltmeter and recorded on a Hewlett-Packard 562 printer, or to a Hewlett-Packard 412A vacuum tube voltmeter, feeding a Hewlett-

Packard 405CR digital voltmeter and a Hewlett-Packard 561B printer. Readings were made at intervals of from 10 seconds to 100 seconds depending on the rapidity of the temperature decay.

All surfaces of Chamber I visible to the specimen were coated with platinum black to reduce their reflectivity to a minimum. Because of the difficulty in depositing good coats of platinum black over large areas, portions of the interiors were originally painted with Parson's black paint, but some of the early data aroused questions as to the blackness of the chamber. The Parson's black was therefore stripped off and techniques developed for depositing platinum black over the interior walls. The change had no apparent effect on the data recorded, as is shown in the data curve.

DEPOSITION OF PLATINUM BLACK COATINGS

Although it is a little aside from the main purpose of this report, a short discussion of the art of preparing platinum black coatings appears desirable at this point. Platinum black is, basically, a very poor platinum plating. It has been used for more than 60 years as a non-polarizing coating for electrodes in electrochemistry, and for a number of years as a coating for receivers in the measurement of radiant energy. Properly prepared, it is an almost perfectly black coating which is stable to temperatures above 500°C in air but which is quite fragile to the touch. Rubbing the surface with a tissue removes a fine black powder and leaves a diffuse grey coating on the substrate. Data taken at NRDL (reference 2) indicate that it radiates as an almost perfect diffuse radiator with no significant deviation from the cosine law, at least at elevated temperatures.

Platinum black, as prepared at NRDL, is electroplated from an aqueous solution of chloroplatinic acid, H_2PtCl_6 , 30g/liter with 0.3g/liter of lead acetate, PbAc_2 , added. The material to be blackened is made the cathode (negative) terminal with a platinum anode. The distance from the anode to all parts of the cathode should be approximately the same, requiring a shaped anode in many cases. The conical shields in Chamber I were blackened in a platinum crucible, where the crucible held the solution and was the anode. Current densities are in the range of 0.1 to 0.5 amperes per square inch. The actual values used are determined by the amount of bubbling which occurs at the cathode surface. The bubbling should be free and abundant, but the bubbles should not be large, since large bubbles may mask portions of the surface from the electric field, and their bursting may knock off portions of the coating. Gentle movement of the piece being coated in the solution helps in removing the bubbles and also tends to even out differences in deposition rate due to variations in the anode-cathode distance.

The reaction has a peculiar, and, at least by the author of this report, not very well understood tendency to reverse at times, and a nearly complete coating can be neatly and rapidly stripped off in an attempt to make it a little better. The reversal usually occurs if the current has been interrupted with both electrodes in the solution, or if the electrodes are immersed and the power turned on. Consequently, the voltage should be applied to the system with one of the electrodes out of the solution and the circuit completed by lowering the electrode into the plating bath. The reversal may frequently be re-reversed by momentarily increasing the voltage across the bath by 50% or so. A relatively high voltage (up to 25 volts) applied to the bath through a dropping resistor appears to be more effective in minimizing difficulties

than supplying the same amount of current from a constant voltage source. The voltage across the bath is usually less than 10 volts.

The substrate materials to be coated must be immaculately clean. The importance of this cannot be over stressed. The substrates used here were first cleaned of all oxides, greases, dirt, and so forth, with whatever combination of acids, detergents and organic solvents that were effective and then the surfaces were mechanically buffed with an ink eraser in an electric eraser holder such as is used by draftsmen. The gold specimens reported in the data section were boiled in concentrated nitric acid, the copper chambers were cleaned with strong hydrochloric acid and a brief rinse of nitric acid, and platinum specimens (not reported) were flamed to white heat with a torch before the buffing stage. A final dip in clean acetone and a rinse in distilled water was frequently desirable.

The specimen should be removed from the plating bath with the voltage still applied if possible. After a brief inspection to determine the appearance of the coating it may be replaced, but if the removal is to be for more than a few seconds, the coating should be rinsed in running hot water immediately. If the current must be interrupted before the coating can be removed from contact with the solution, the removal must be accomplished as rapidly as possible and the coating rinsed immediately, even before inspection. When the coating is finally satisfactory it should be rinsed extensively in running hot water and then with distilled water. The specimens may be simply air dried or dried with a stream of warm (not hot) air from a blower, but they should not be exposed to temperatures above 100°C before they are completely dry.

It is extremely difficult to coat to the bottom of grooves or

holes or into corners due to the masking of the electric field by the sides. Chamber I was coated at a rather low current density with a long plating time because the bubbling was too violent for high current densities. The final current density was well below 0.1 amperes/cm^2 and the coating time extended for over one hour.

With some experience, visual inspection of platinum black coatings immediately after drying provides a good indication of their emittance properties. While it is true that the wave lengths involved between those observed with the eye and those at which the specimen radiates are quite different, a good coating appears very dark and diffuse, especially when viewed under a strong lamp. The comparison to black velvet of a good coating reveals differences easily distinguishable. This ability of the eye to evaluate a good platinum black is a valuable tool.

ERROR ANALYSIS

Air Conduction

The residual pressure inside a chamber with walls at L/He temperature is due entirely to helium, since only helium and hydrogen have appreciable vapor pressure at 4°K . This pressure has been estimated to be as low as 10^{-20} Torr (Reference 3) and is almost certainly below 10^{-10} Torr. Since from Equation (2) a pressure of 3×10^{-8} Torr appears to result in negligibly low air conduction at 100°K , conductance at a pressure of 10^{-10} Torr would be negligible even if equation (2) should prove to be considerably in error.

When liquid nitrogen was used in Dewar I, as it was at some

of the higher temperatures, the vacuum was probably 5×10^{-5} Torr or higher. Because of the long conduction path from the chamber to the pump, it took 2-3 days of pumping to reduce the pressure to its minimum value. This amount of time was normally not available so measurements with L/N were abandoned for the later experiments. The cryogenic pumping with L/He was very fast and negligible pressures were reached by the time the dewar was full. A pressure of 5×10^{-5} Torr is not high enough to result in significant errors near 300°K , as is shown in Fig. 5, where the rate of decay of a polished gold button is plotted against pressure, but is certainly not low enough for the lower temperature measurements. This was shown by the difference in curves when data was taken at the same temperature with L/N and L/He in the dewar. These data are shown on some of the data curves.

Support Conduction

The effect of support conduction cannot be estimated from equation (4) since data are completely lacking on the thermal conductance of alumel and chromel below 300°K . The thermal conductance of the thermocouple materials near 300°K is near that of monel, on which data is available, and presumably follows the same type of curve at lower temperatures. The thermal conductivity of monel decreases by a factor of 20 in going from room temperature to 4°K .

To evaluate the loss experimentally, a gold specimen coated with platinum black was suspended from 5 mil (.005") and 1 mil (.001") wires and the rates of temperature decay recorded. Figure 6 shows that there was no difference beyond experimental error. As a change of 25 times in the cross sectional area of the supports results in an unmeasurable difference, it may be assumed that the conduction loss down the supports is negligible.

The very low thermal conduction error is due to the very high thermal resistance of the alloy wires near 4°K . Figure 9 shows the effect of support conduction with the walls at L/N temperatures when 5 mil wires are used. These data were not taken in Chamber I or II but are included to show the effect. This effect of the conduction is to make the apparent emittance too high, and Figure 7 is plotted in terms of the emittance correction necessary for a $3/4$ inch diameter button. One mil diameter wires would have resulted in an error of less than .01 over the entire range of Figure 7.

Figure 8 presents the emittance of a polished gold specimen as measured in Chamber I using L/He cooled walls and one mil thermocouple wires. Since the total rate of heat loss from this specimen represents an emittance of 0.02 for its surface, the contribution of air and support conduction cannot exceed this value, even if the emittance of gold were zero. The theoretical emissivity of gold at 100°K from the equation of Davisson and Weeks^{3,4} is $0.75(\text{pT})^{1/2}$, or about 0.007. Bulk gold cannot be polished to the degree possible with a harder, more amorphous material such as glass, and the actual emittance of this particular specimen is probably higher than the theoretical value. The total error in the measured emittance values introduced by air and support conduction may therefore be stated to be not more than 1% of the emittance of a black surface at 100°K . That the actual value of error is even smaller than this is evidenced by the straightness of the temperature vs emittance line in Fig. 8. Any appreciable conduction loss would result in a curvature of the line, with a decreased slope at lower temperatures.

Chamber Blackness

The energy radiated by a black wall at 4°K and absorbed by a

perfectly black specimen at 80°K is less than 1 part in 10⁵ of the energy the specimen is radiating, and may be considered zero for all practical purposes. The effect of the energy reflected back on the specimen may be estimated from Equation (23). Assuming, in accordance with the immediately preceding discussion, that all the terms in Equation (23) except the first and last are negligible, we may state that

$$Q_t = mCT_s = A\epsilon_1 \sigma T_s^4 - \frac{A_1 \epsilon_1^2 \sigma T_s^4}{\frac{A_2 \left(\frac{\epsilon_2}{1-\epsilon_2} \right)}{A_1} + \epsilon_1} \quad (24)$$

The value of emittance, ϵ^* , which is recorded in the data curves is calculated as

$$\epsilon^* = \frac{mCT_s}{A\sigma T_s^4}$$

but, from Equation (24) it can be seen that actually

$$\epsilon^* = \epsilon_1 - \frac{\epsilon_1^2}{\frac{A_2 \left(\frac{\epsilon_2}{1-\epsilon_2} \right)}{A_1} + \epsilon_1} \quad (25)$$

The ideal way to prove that a chamber is black is to measure the emittance of a perfectly black specimen and obtain $\epsilon^* = 1.0$. From the definition of emittance it follows that no emittance can exceed one, and if a measured value of $\epsilon^* = 1.0$ then the second term of Equation (25) must be zero. Unfortunately, there are no materials with an emittance of one. Fig. 9 gives the emittance of platinum black vs temperature, and we have found nothing blacker. Proof of the blackness of the chamber requires more indirect evidence.

The first measurements of the emittance of platinum black were made with the chamber coated with Parson's Black paint, with the

results shown in Fig. 9. As there was some doubt as to the emittance of Parson's Black at low temperatures, the point was stripped off and the chamber re-coated with platinum black, as described in the Experimental Apparatus section. Comparison of Fig. 9 and 10 indicates that the platinum black wall had a higher emittance than the Parson's black wall at L/N temperature, but the data points obtained with the two wall coatings were identical, indicating that the chamber emittance is little affected by changes in the wall emittance of 50% or so. This is possible only if the chamber is effectively black, or nearly so, even with the lower emittance wall coating. With the chamber coated with platinum black and a platinum blacked specimen measured, $\epsilon_1 \approx \epsilon_2$ and Equation (25) can be simplified to

$$\epsilon = \frac{\epsilon^* (1 + \frac{A_1}{A_2})}{\frac{A_1}{A_2} \epsilon^* + 1}$$

Substituting the value for ϵ^* obtained at 77°K for a 3/4 inch platinum black specimen,

$$\epsilon = \frac{0.72 (1 + \frac{1}{25})}{(\frac{1}{25}) 0.72 + 1} = 0.728$$

so that the value of emittance ϵ^* reported could have been 1% too low in this rather small chamber with a wall area only 25 times the specimen area.

To increase the ratio A_2/A_1 a button 3/8 inch in diameter was coated with platinum black and its emittance determined with results as shown in Fig. 9. The minimum value of 0.92 for the emittance obtained at 77°K was considerably above that of the 3/4 inch specimen. To account for this different value in terms of chamber reflectance required that the emittance of the walls of the chamber, ϵ_2 , be less than 0.1, a value

that appeared impossibly low for a platinum black or Parson's black coated wall and a result completely at odds with the effect of changing the wall coatings. A second experiment was therefor run using 3/8 and 3/4 inch diameter buttons coated with 3M Velvet Black (Fig. 11). In this case there was no difference between the two sizes of specimens and the difference in the platinum black specimens was attributed to an actual difference in the two platinum black coatings. This is discussed later, in the results section.

The effect of internal reflection in Chamber I was considered negligible, causing less than a 1% error at 77°K in the measured value of emittance of a 3/4 inch diameter specimen of high emittance.

Chamber Accuracy

The evidence given so far in this section indicates that air conduction, support conduction and wall reflection and radiation effect the measurements by less than 1%, and all terms in Equation (9) except the first may be neglected and at least for Chamber I,

$$Q_t = A\epsilon_1\sigma T_s^4$$

with an error of less than 1%.

Instruments

The determination of temperature is always difficult when a high degree of accuracy is required. Thermocouples are the most flexible and simple of temperature measuring devices, and in the

experimental apparatus used here they are the only practical sensor, but to obtain accurate measurements demands a considerable number of experimental precautions. Chromel-alumel thermocouples (Type K) were chosen because of their low thermal conductance and their considerable physical strength. Other combinations have a somewhat higher thermal emf, but the difference is not enough to make any change in the type of voltmeters required to measure the thermocouple output. Like all thermocouples, the emf temperature curve of chromel-alumel couples varies somewhat in each batch of wire, in different portions of the same batch, and with the physical treatment the couple experiences. The National Bureau of Standards has published a table of thermal emf vs temperature (Reference 5) for type K chromel-alumel thermocouples, with the recommendation that individual couples be calibrated and linear corrections be applied to correct for the deviations that are always present. It is interesting to note that NBS has actually published two tables (References 5 and 6) which differ from each other by about 2% at L/N temperature when using a 273°K reference junction. The emf measured is, of course, the difference between two thermocouple junctions, one at a reference temperature and one at the unknown temperature, and the reference junction must be held stable to a higher degree of precision than that required of the measurement of the unknown.

The initial measurements at NRDL were made using a 296°K (23°C) reference junction which was thermostated and held stable to $\pm 1/4^\circ\text{K}$, but because this error at 296°K represents an error of about $\pm 3/4^\circ\text{K}$ at L/N temperatures, the system was revised to use a L/N reference junction. Each individual couple was calibrated in place in the closed and evacuated chamber by filling the dewar with L/N in the L/He section and allowing everything to come to equilibrium. We had expected that the one mil wires would be rather variable, as they were not only very small but under tension (a 6 g specimen loads the wires to

about 600 kg/cm²), but this did not prove to be the case and the one mil wires were no more variable than larger wires, and the tension did not appear to affect the emf measurably. This emf fell between the two NBS values, and was very reproducible between different specimens.

The actual temperature is probably known to about 0.5°K at L/N temperature, with an error which may be somewhat larger at other temperatures, where calibration was not possible. The error in temperature determination is in all cases less than 1%, however.

The actual parameter measured in the experiments was the rate of change of temperature at a given temperature, and

$$Q_t = mCT$$

The mass may be measured on a conventional analytical balance to an accuracy of better than 0.1%. The heat capacities have been taken from published data (Reference 7,8,9) for pure metals. These values are given to three significant figures but it is doubtful if they are actually known to 1%, particularly since C varies rapidly in the temperature range from 77°K to 300°K for all metals, and an error of 1°K in temperature can result in an error of more than 3% for some metals, such as aluminum. To minimize this latter effect, gold substrates, which exhibit the smallest change in C from 77°K to 300°K, were used wherever possible. The mass of the coating, in the case of the coated specimens, was never more than 1 or 2% of the total weight, but because of the probably higher heat capacity of the coating than the substrate, the error from this source might be as high as 5% (This error would always give a low emittance value).

The rate of change of temperature with time is extremely low with samples weighing several grams near 77°K, requiring 1000

seconds or more to change 1°K even for a black specimen. This represents a rate of change of less than 0.016 microvolts per second and requires a very low drift rate in the measuring voltmeter to measure accurately.

All voltmeters exhibit a certain amount of jitter, and this may represent $\pm 0.3^{\circ}\text{K}$ or better even for the very high quality units used here. The readings are taken at frequent (10-100 seconds) intervals and replotted on an expanded scale to determine the exact slope but the final accuracy of the slope determination is probably not better than $\pm 5\%$.

The errors are not completely random. Air and support conduction losses tend to give a high value of emittance, while errors due to the coating heat capacity would tend to give low values. Contamination of the L/N reference with oxygen, the most likely error, would result in too low an emf being measured for the temperature of the specimen, and a high value of emittance. The voltmeter jitter is random. Adding everything up, the accuracy of the data is within $\pm 10\%$.

RESULTS

A survey, by letter, of the requirements for materials of various groups and companies in industry who were interested in the passive cooling of infrared detecting systems in space was made at the beginning of this project. The materials tested were chosen, as far as possible, from the results of this survey. It was not possible to test all the materials requested. Platinum black coatings were not mentioned in any of the replies to the survey, but was added both to test the system and because experience at NRDL has shown that it is one of the

blackest of materials.

The data are presented in graphs from Figure 9 on, with artist's drawings of the appearance of many of the black coatings under 500X magnification. Drawings are used rather than photographs because although the various coatings appeared different under a microscope, it was impossible to obtain a photograph that showed the difference. The reason was apparently the shallow depth of field of the microscope, which could not simultaneously focus on the entire depth of the coating. Drawings can also somewhat exaggerate those features which are clearly evident, but which do not reproduce well unless they are exaggerated.

Metal Blacks

Platinum Black.

The technique of coating a specimen with platinum black has been discussed previously in the experimental section. The results of the emittance measurements, some of which were considered in the section on chamber blackness, were not as expected. Careful consideration of the specimens led to the conclusion that the coating on the $3/8$ inch button was different from that of the $3/4$ inch specimen, and that the probable cause of the difference was a different current density during the coating process. Because of limitations in the coating system, the total amount of current which flowed through the $3/8$ inch button was nearly the same as that through the $3/4$ inch button, leading to a higher current density for the smaller specimen. The holder which held the specimen in the path and which formed the electrical conductor was then modified and a very heavy coat was laid down on a $3/4$ inch button, at a current density similar to that used for the $3/8$ inch button. The

result, also shown in Fig. 9 was that the emittance of a heavy coating of platinum black was lower than a light coating.

After determining that there was no experimental error, and that the data were real, a tentative explanation was worked out for the apparently contradictory results. Electron diffraction analysis has indicated that the interface between platinum black and copper may contain a copper-platinum alloy (Reference 10), and that a similar state exists for platinum black on silver. It is probable, therefore, that a gold-platinum black interface is also complex, in a manner that would affect its emittance characteristics. A very heavy coat of the black, however, would mask this interface, and would, essentially, be a coating of platinum black on platinum. Scratching the heavy coat gave some indication of specular platinum under the black. Some early data taken during this project seemed to indicate that the emittance of platinum black on a platinum specimen was lower than that on a gold specimen. These results were not reported in detail because the geometry of the specimen was different from the buttons used for the other experiments, and the results are not strictly comparable, but the data within its limitation tends to support the explanation given above.

Gold Black.

Three-quarter inch diameter gold buttons were coated with gold black by evaporation of gold from a tungsten heater in an atmosphere of argon at a few microns pressure in a manner similar to that described in Reference 11. Thin and thick coatings were prepared and the data are presented in Fig. 12. The emittance of this material in the thicknesses measured was not as high as that of platinum black,

although increasing the thickness increased the emittance. The coating was extremely fragile and had to be handled with extreme care.

Black Paints

General

A uniform coat of paint on a small metal disc is difficult to form, even though a larger area can be sprayed or painted with an apparently uniform coat. Several methods were used for the paints reported here, none of which were completely satisfactory when viewed under a magnification of 100X, where they exhibited a mottled or non-uniform surface. An attempt to show this mottling is made in the drawings. Except for the Parson's Black, it was not necessary to use undercoatings to make the paint stick.

The amount of paint on the surfaces of the specimens, even when there is no visible difference in the appearance, effects the low temperature emittance. Several attempts were made to measure the thicknesses of the coatings, all of which were somewhat ambiguous. The easiest method, and one which is reported for all the specimens, was to determine the increase the weight of the specimen when it was painted. This measurement does not, however, give any value for the variation in thickness of the coating across the buttons, nor the different thickness on the edges. The use of feeler gauges or micrometers gives definite values which can be read fairly easily, but they put a pressure on the coating, causing an unknown amount of compression. Also, the coatings have ripples in them on a scale which is very small compared to the anvil of a micrometer and which is large compared to the wavelength of the emitted radiation. The micrometer measurement gives the peak

values of these ripples (or graininess) which may not be the same effective value seen by the radiated energy.

A couple of the coatings flaked on the gold substrate when they were heated. These flakes were mounted edge-up under a measuring microscope and the thickness measured. Parson's Black painted on glass could be flaked off and measured similarly. This method suffered the same limitations as the weight measurement, in that it did not give the range of values for different areas, and when deposited on a glass substrate, the film was a different one than the one actually measured. The density of the dried films, as far as could be determined, was between one and two.

All thickness measurements on the paints when they could be made on the same coating, agreed within about 25%, and this is the accuracy believed applicable to the values for thickness given on the data curves. The paints tested, their manufacturers and the method of application were as follows:

Parson's Optical Black Lacquer, Thos. Parson's and Son, LTD, Surrey, England (Fig. 10).

This paint was diluted with acetone and applied in small drops to the center of a gold button spinning at 1800 rpm in a jig. The button was first coated in the same way with a very thin covering of the recommended undercoating, obtained from the same manufacturer. When the dilution of the paint was correct the drops spread uniformly across the button and formed a smooth, dusty black film. Some of the excess paint formed little hard lumps at the edge of the button, which were chipped off after drying. The paint was air dried for several hours before use in the chamber.

Minnesota Mining and Manufacturing Co., Velvet Coating,
9564 Black, (Fig. 11).

This was a thixotropic paint of gel-like consistency, which could be liquified by agitation. The viscosity of the paint depended on the violence of the agitation. The buttons were coated by dipping them into paint that had been reduced to as low a viscosity as possible by stirring with an electric stirrer. The coatings were air dried, and were quite rugged. This was the easiest paint of all to apply.

W. P. Fuller & Co., Fuller Paint, (Fig. 13) Metal Etching
Primer 3811 Black

This paint was thinned with the prescribed thinner and applied with a small brush. It formed a very adherent coating on air drying. It formed the thinnest film of any of the paints, but had an emittance higher than the heavier Fuller enamel.

Fuller Plastic Enamel Velvet Black 1518

This paint was thinned with xylene and applied with a brush. The button coated with this paint remained in the dewar at L/N temperatures for about 5 days with the vacuum system running continuously. There was no difference in the appearance of this, or any, paint after the measurement from the appearance before the measurement.

Midland Industrial Finishes Co., Waukegan, Ill. (Fig. 14)

Sicon Black 7X942

This paint was diluted about $1/3$ with the recommended thinner and applied to the button by dipping. The coating was air dried for about an hour and then baked at 200°C for 30 minutes. It formed a heavy coating that was quite hard.

Anodized Aluminum (Fig. 15,16,17,18).

These coatings were made by J. H. Weaver of ASD, USAF, and were formed by electrolytic oxidation of pure aluminum in sulfuric acid. The thicknesses were measured at NRDL, on other samples than the one run, by cutting the specimens through the coatings, carefully polishing the edges and looking at them, edge on, with a measuring microscope. The thicknesses were quite uniform and easily measured. The samples looked like highly polished pieces of metal, with the coating quite transparent to the eye.

All four of the coatings showed a maximum emittance of about 230°K - 250°K where the peak wavelength of the radiated energy in air is 12 microns. The fall off in emittance as the temperature increases is presumably due to the transparency of sapphire to radiation of wavelength shorter than six microns, and at lower temperatures to the decreased thickness of the coating as compared to the peak wavelength of the radiation.

CONCLUSIONS

The highest emittance coating measured was platinum black

deposited electrolytically on bulk gold. An excessive thickness of this coating appears to reduce the emittance, perhaps because the coating forms a gold-platinum alloy at the interface of the gold and the platinum black, an interface that is masked by a heavy coating of the black. It is probably safe to use these data for similar coatings on copper and silver, and other noble metals. Because of the method of preparation coatings similar to those on gold would be difficult to form on metals electropositive with respect to hydrogen, such as aluminum and iron. The coating would also be difficult to apply to very fragile materials.

The extreme fragility of the gold black coating makes its use undesirable when it can be avoided. The method of deposition, however, enables it to be deposited on almost any material, even very light ones.

The emittance at low temperatures of the black paints appear to be more a factor of the thickness of the coating than of the specific paint. They are all dispersions of ceramic or carbon black pigments in various organic vehicles, and while variations in the composition of the vehicles or binders may cause considerable change in the physical properties of the paint, the range of variations is not great enough to significantly affect the emittance below 300°K.

The anodized aluminum coatings have the interesting property of a high low temperature emittance with a high reflectivity for visible light. This would be of value when the effect of sunlight must be considered in the design of a system.

The technique of using the rate of change of temperature of a specimen suspended in a vacuum is a valid method for obtaining the

total hemispherical emittance of the specimen to temperature at least as low as 77°K. In a properly designed and operated chamber the emittance of a specimen may be represented as $mCT/A\sigma T^4$ with no error greater than 1% of the emittance of a black specimen at 77°K.

Instrument errors and uncertainties in the heat capacity measurements and temperature determinations, however, reduce the overall accuracy of the data reported here to $\pm 10\%$.

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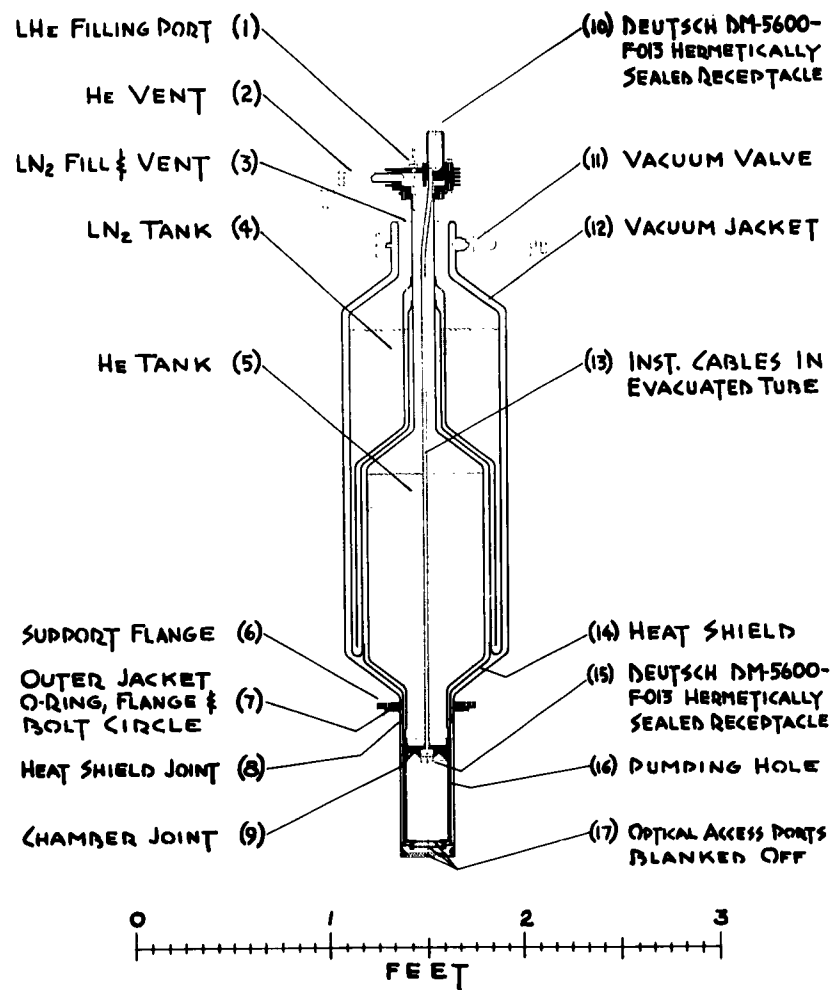


Fig. 1 Schematic drawing of Dewar I.

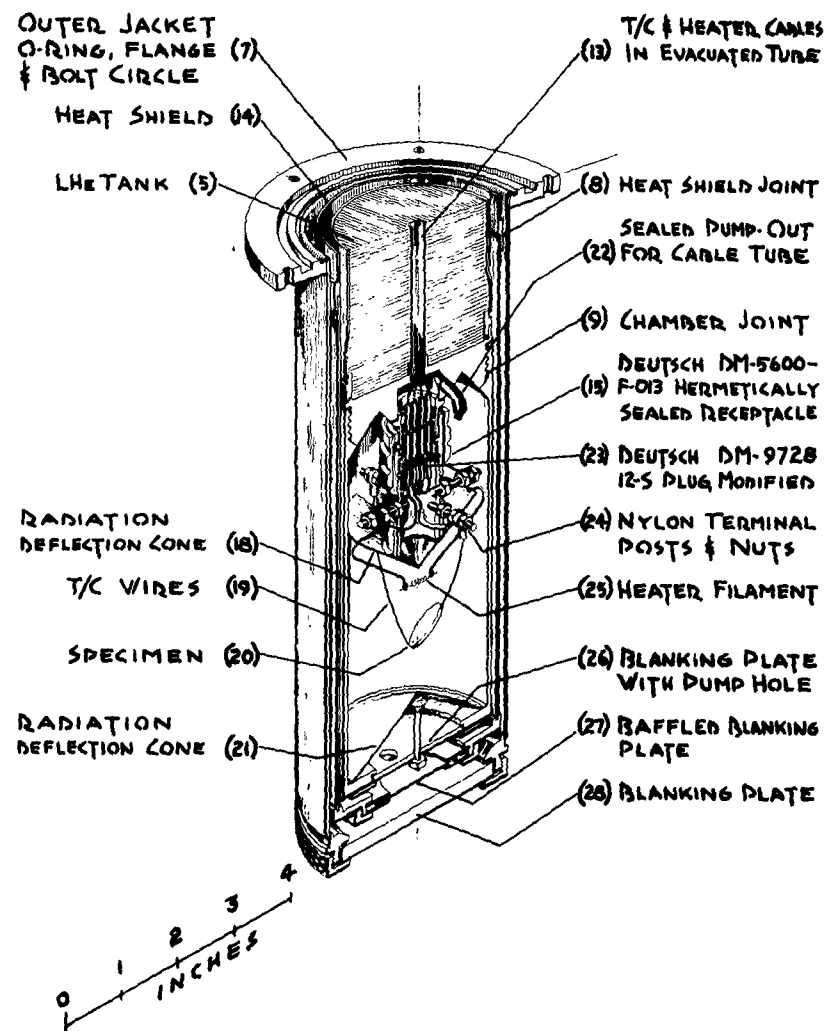


Fig. 2 Schematic Drawing of Chamber I.

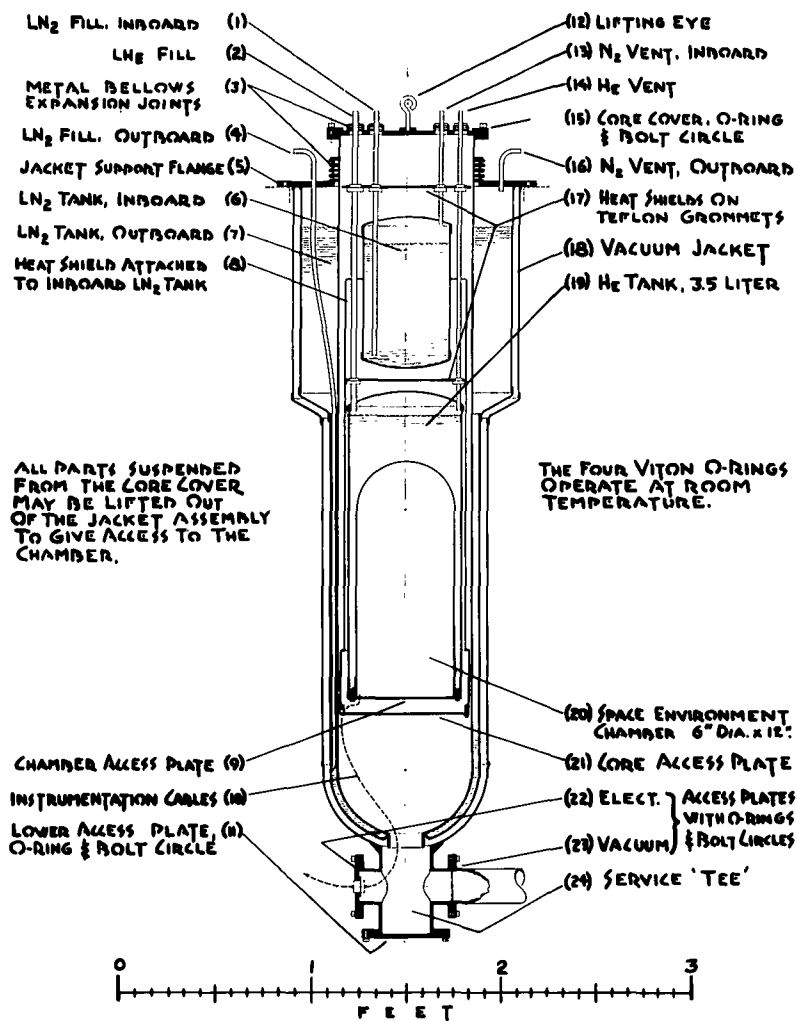


Fig. 3 Schematic drawing of Dewar II.

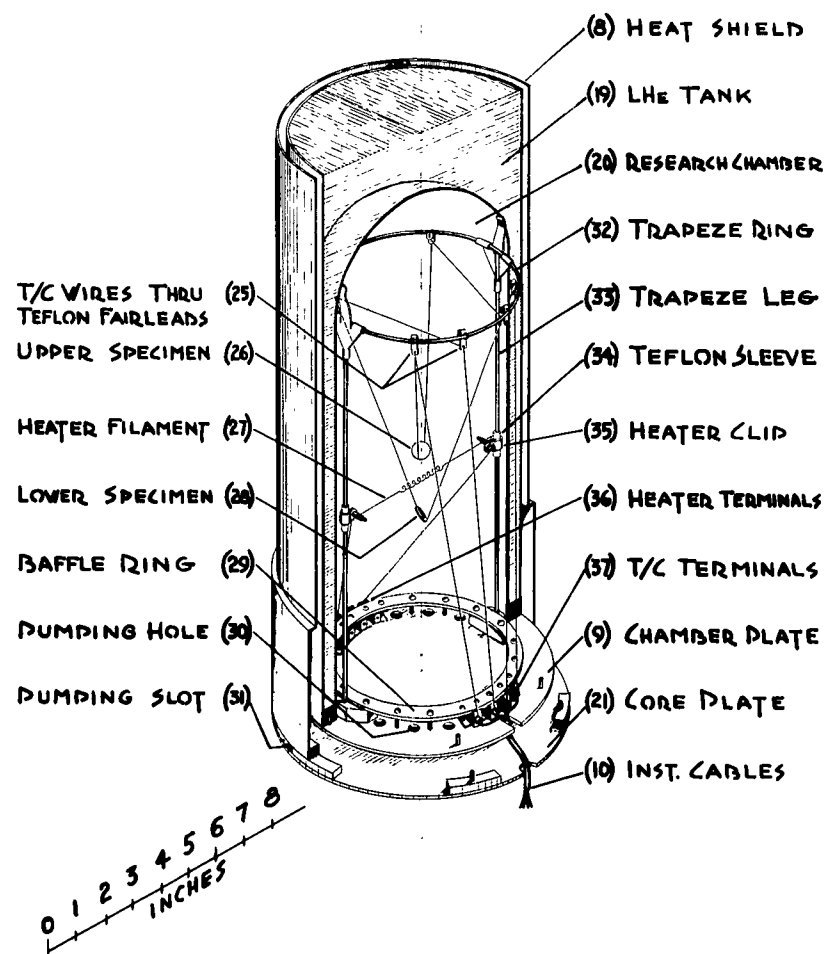


Fig. 4 Schematic drawing of Chamber II.

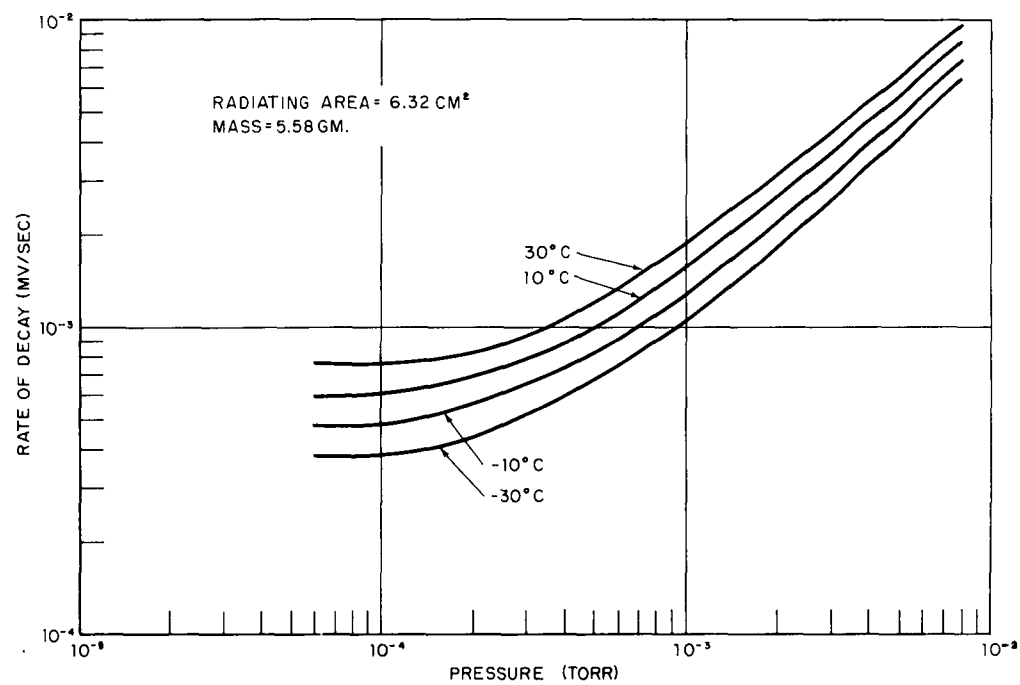


Fig. 5 Effect of pressure in vacuum chamber on the ratio of temperature decay of a polished gold specimen.

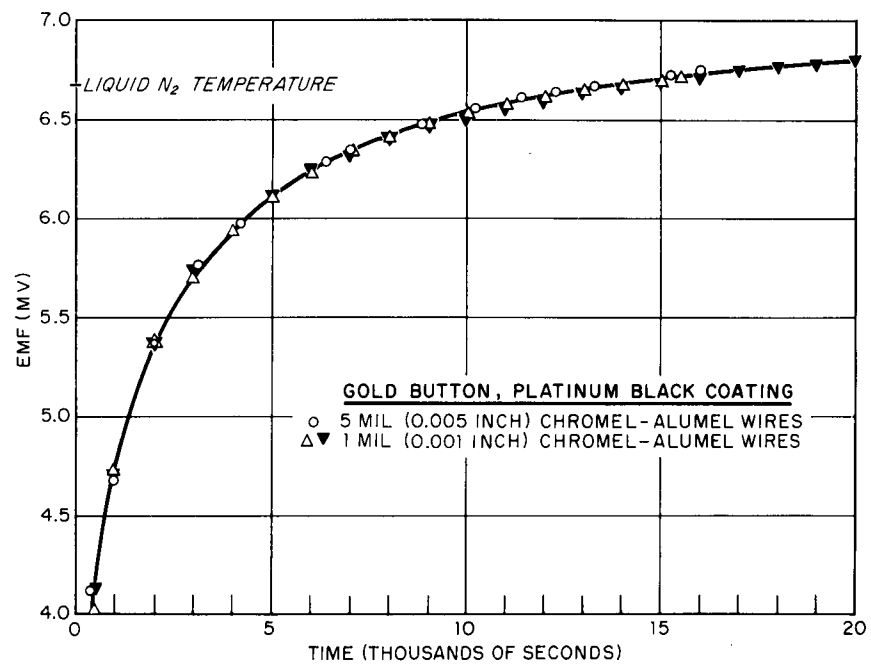


Fig. 6 Effect of changing wire size on rate of temperature change in Chamber I. Walls at L/He temperature.

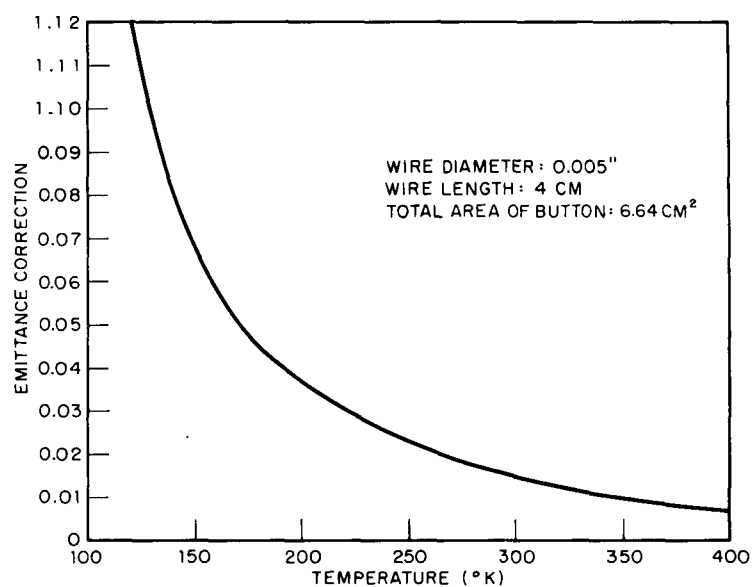


Fig. 7 Apparent emittance due to supporting wires. Data not taken in Chambers I or II, but included to illustrate effect. Walls at L/N temperature.

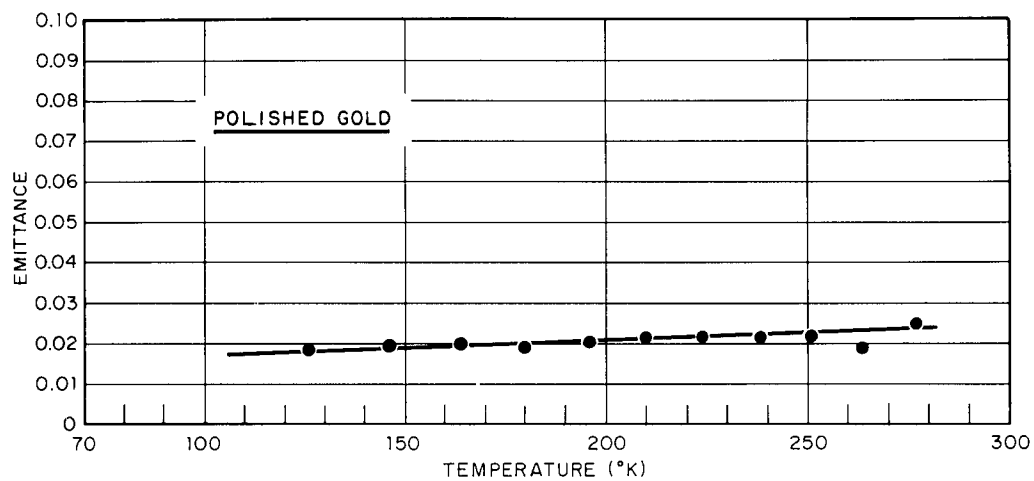


Fig. 8 Low temperature emittance of polished gold.

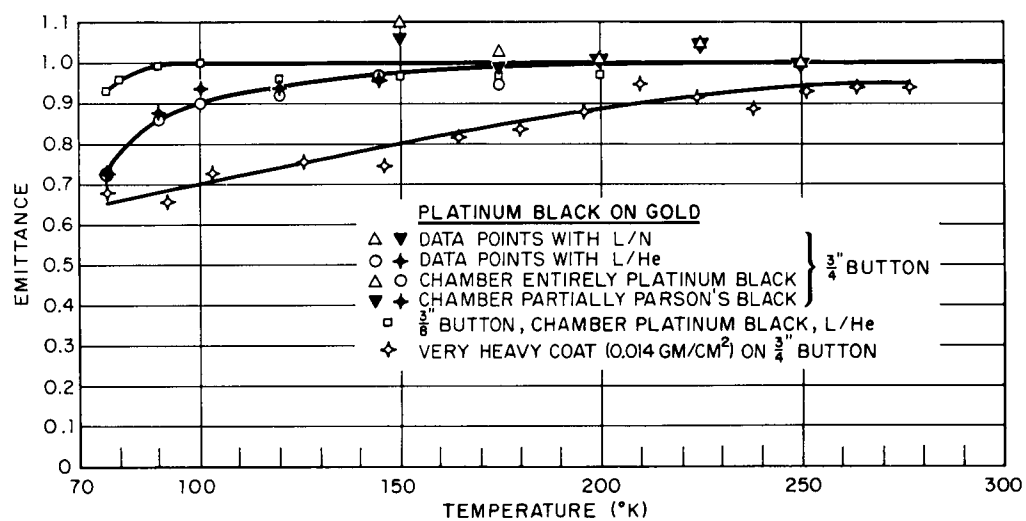


Fig. 9 Low temperature emittance of platinum black on gold.

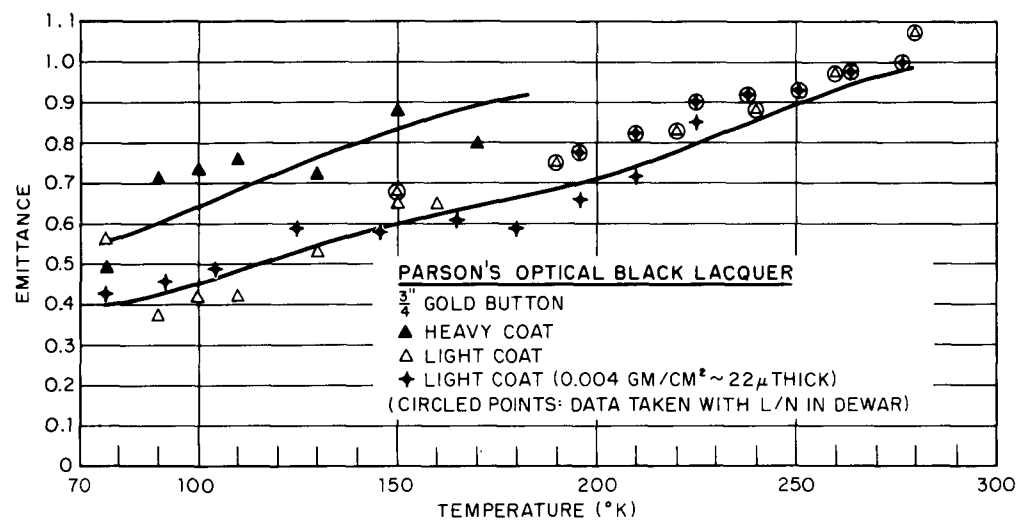


Fig. 10 Low temperature emittance of Parson's optical black lacquer.

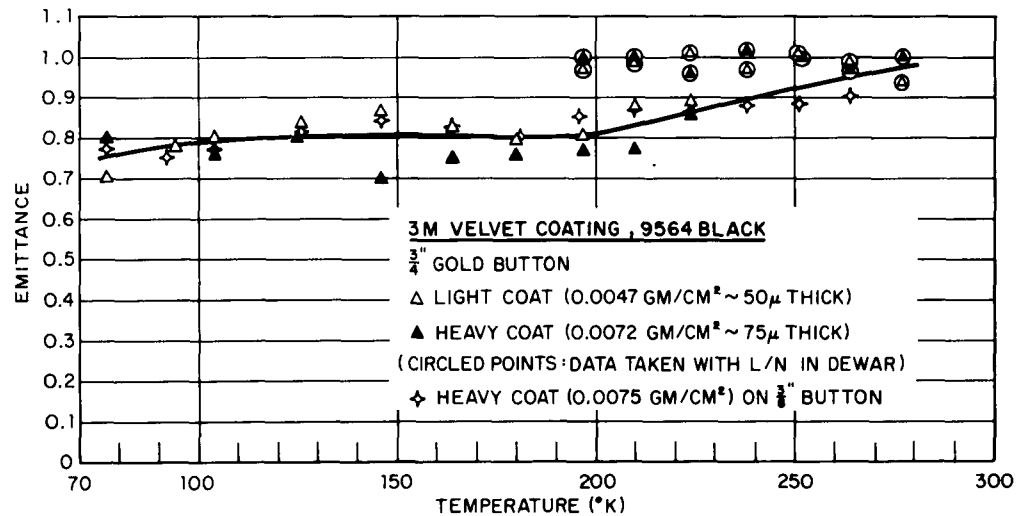


Fig. 11 Low temperature emittance of 3M velvet coating, 9564 black.

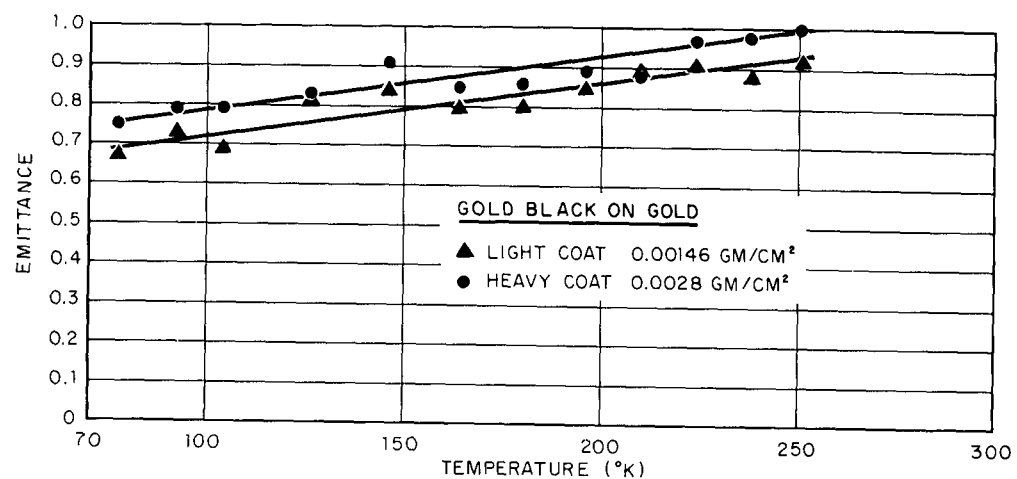


Fig. 12 Low temperature emittance of gold black on gold.

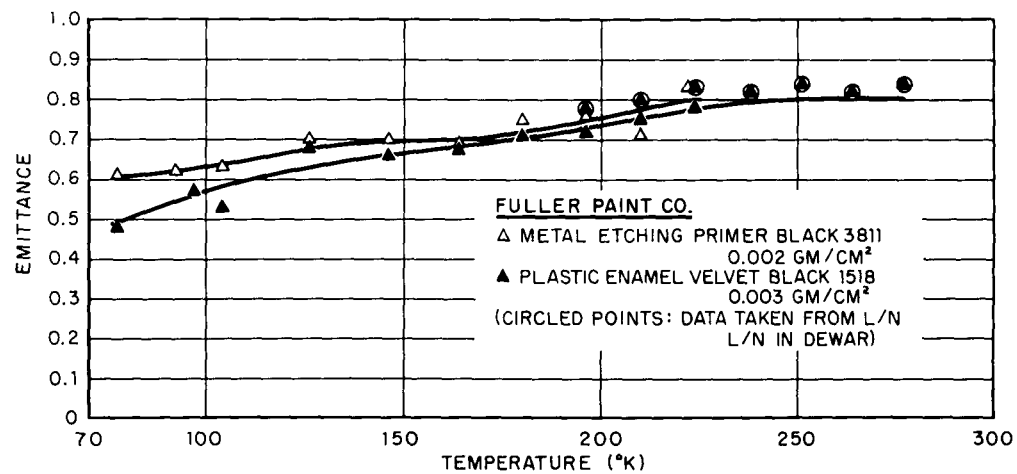


Fig. 13 Low temperature emittance of Fuller Paint Co.

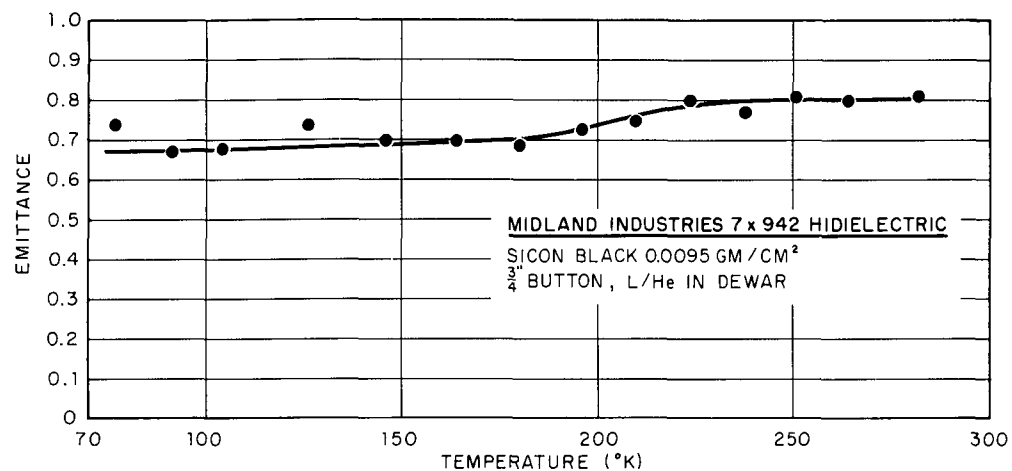


Fig. 14 Low temperature emittance of Midland Industries 7 X 942 Hidielectric.

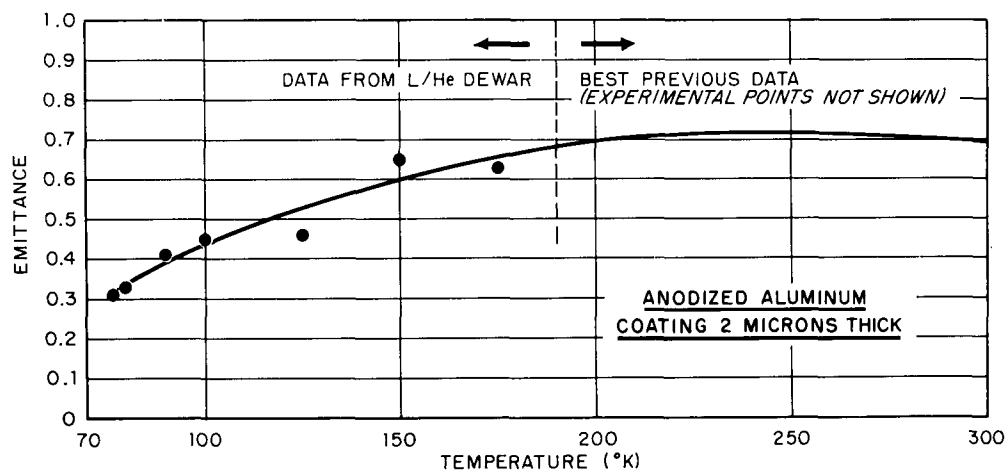


Fig. 15 Low temperature emittance of anodized aluminum coating 2 microns thick.

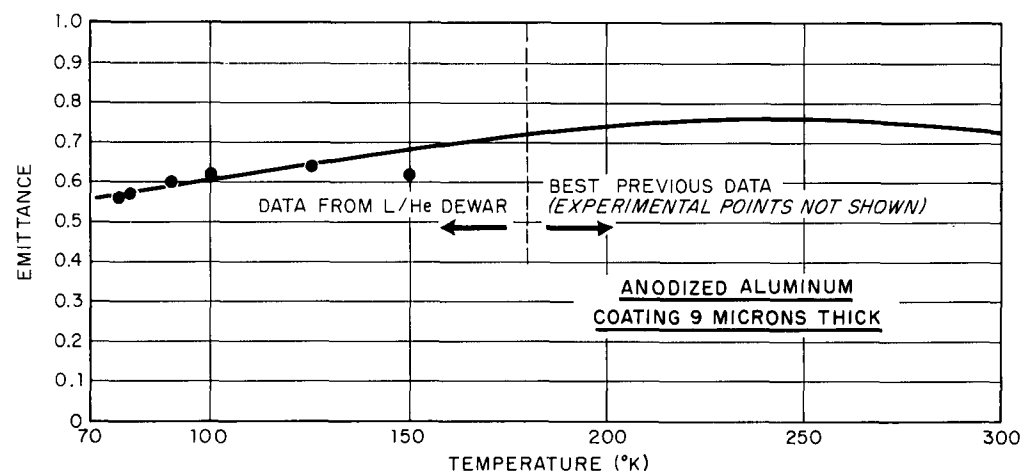


Fig. 16 Low temperature emittance of anodized aluminum coating 9 microns thick.

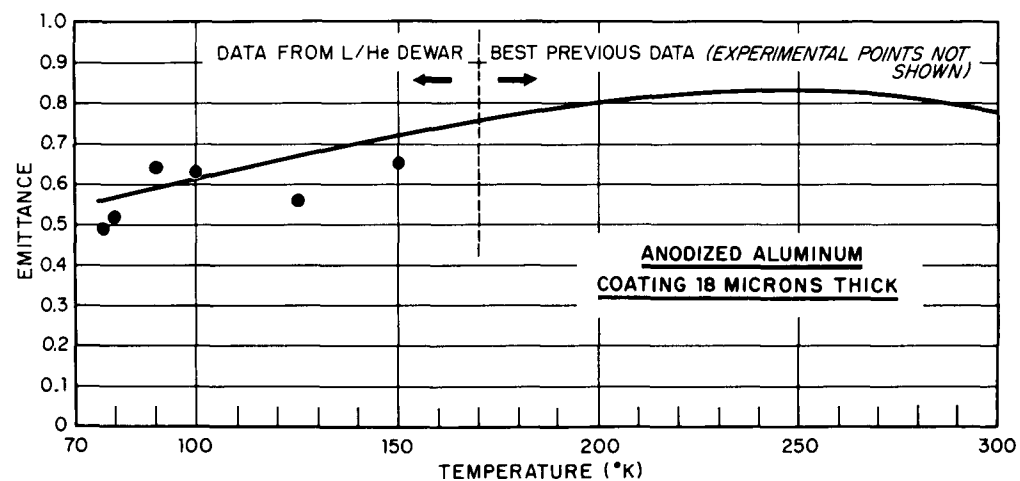


Fig. 17 Low temperature emittance of anodized aluminum coating 18 microns thick.

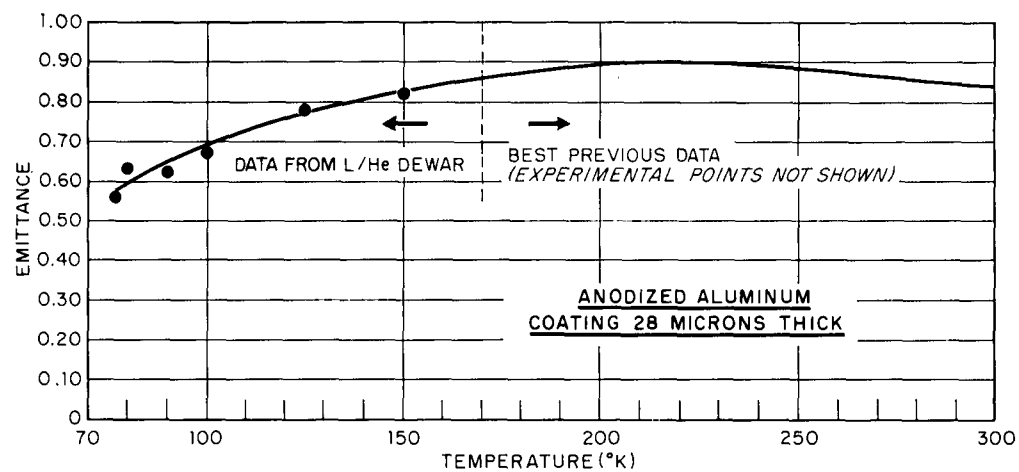


Fig. 18 Low temperature emittance of anodized aluminum coating
28 microns thick.

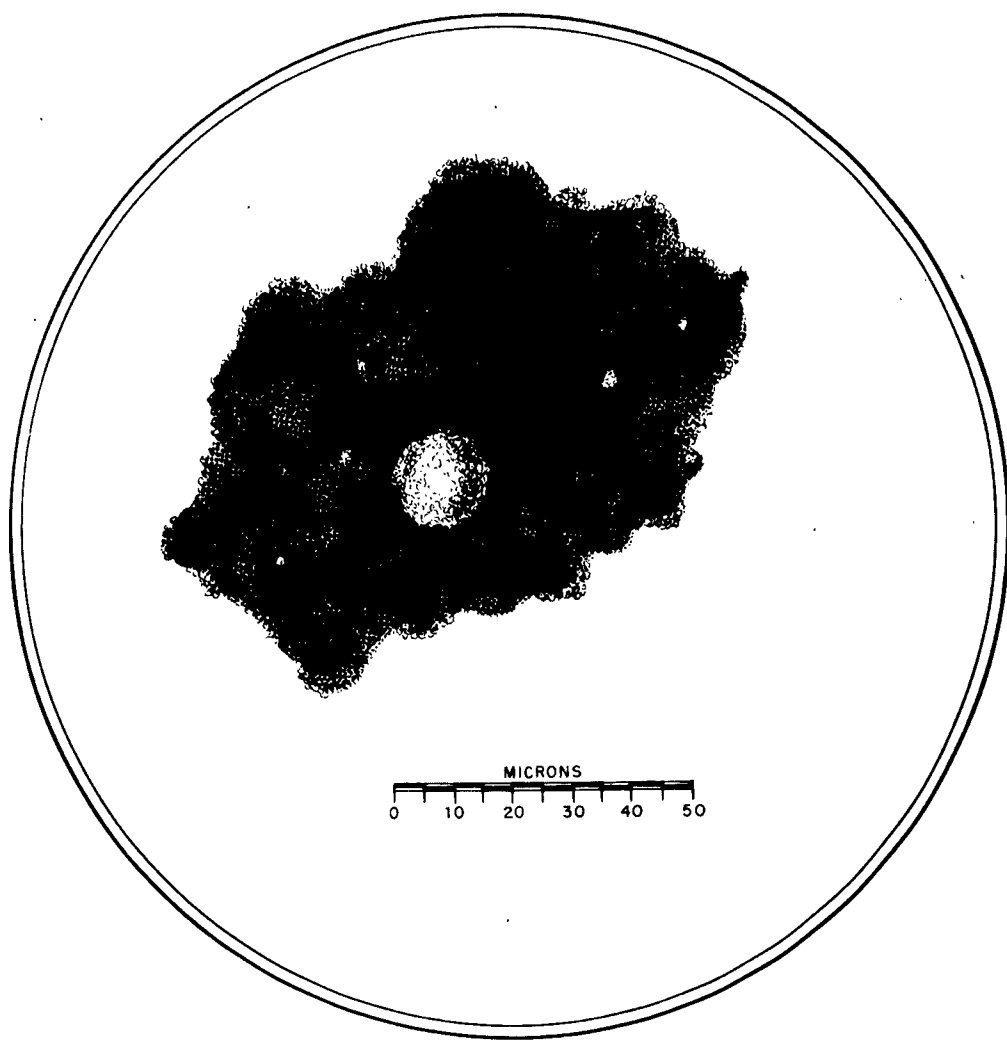


Fig. 19 Artist's drawing of microscopic (X500) appearance of platinum black.

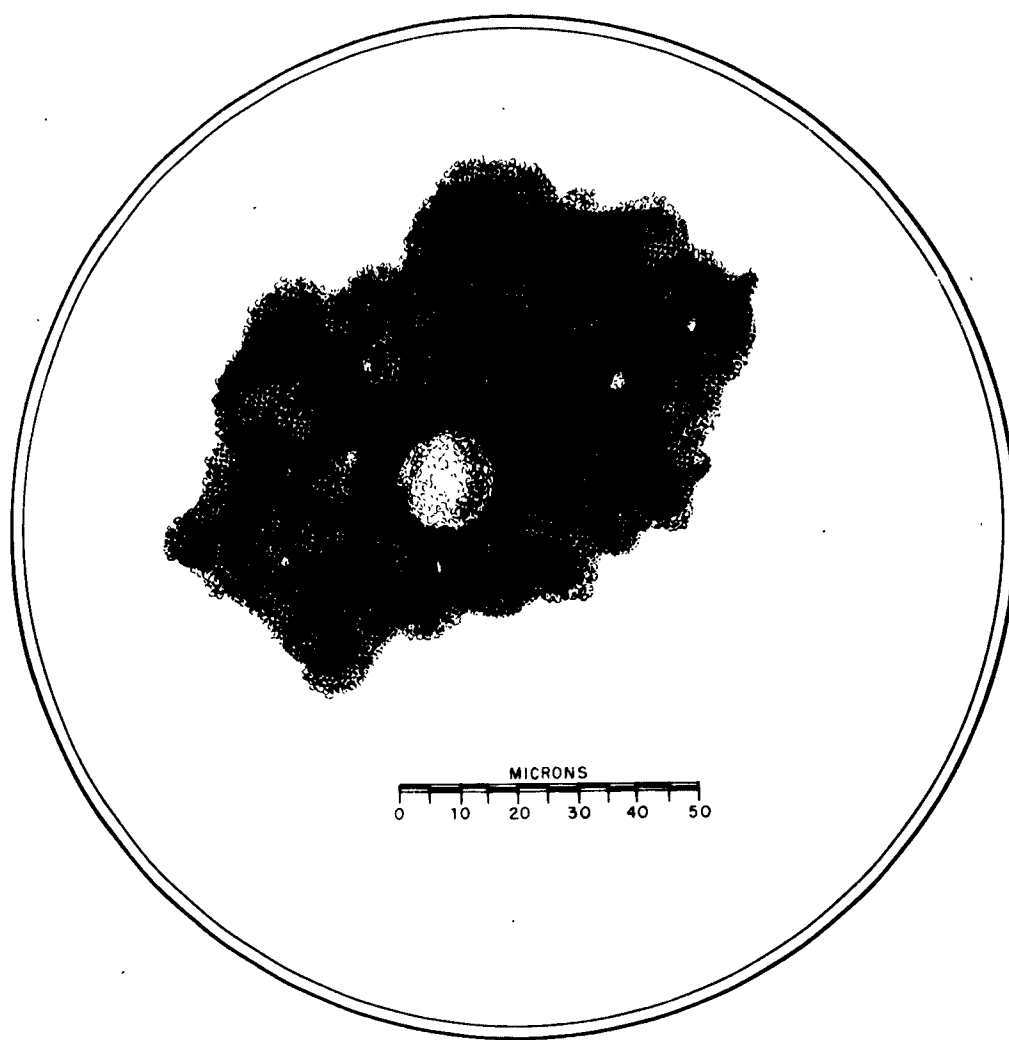


Fig. 19 Artist's drawing of microscopic (X500) appearance of platinum black.

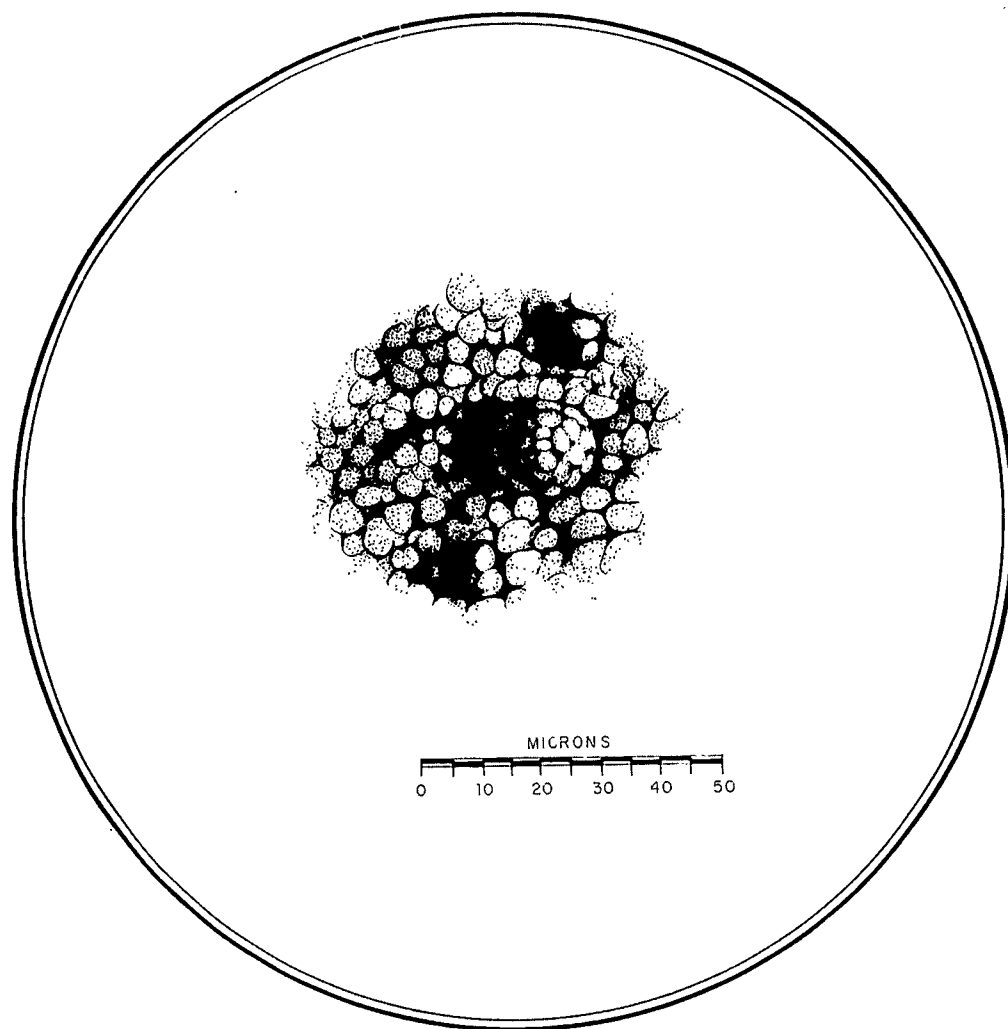


Fig. 20 Artist's drawing of microscopic (X500) appearance of platinum black, heavy coat.

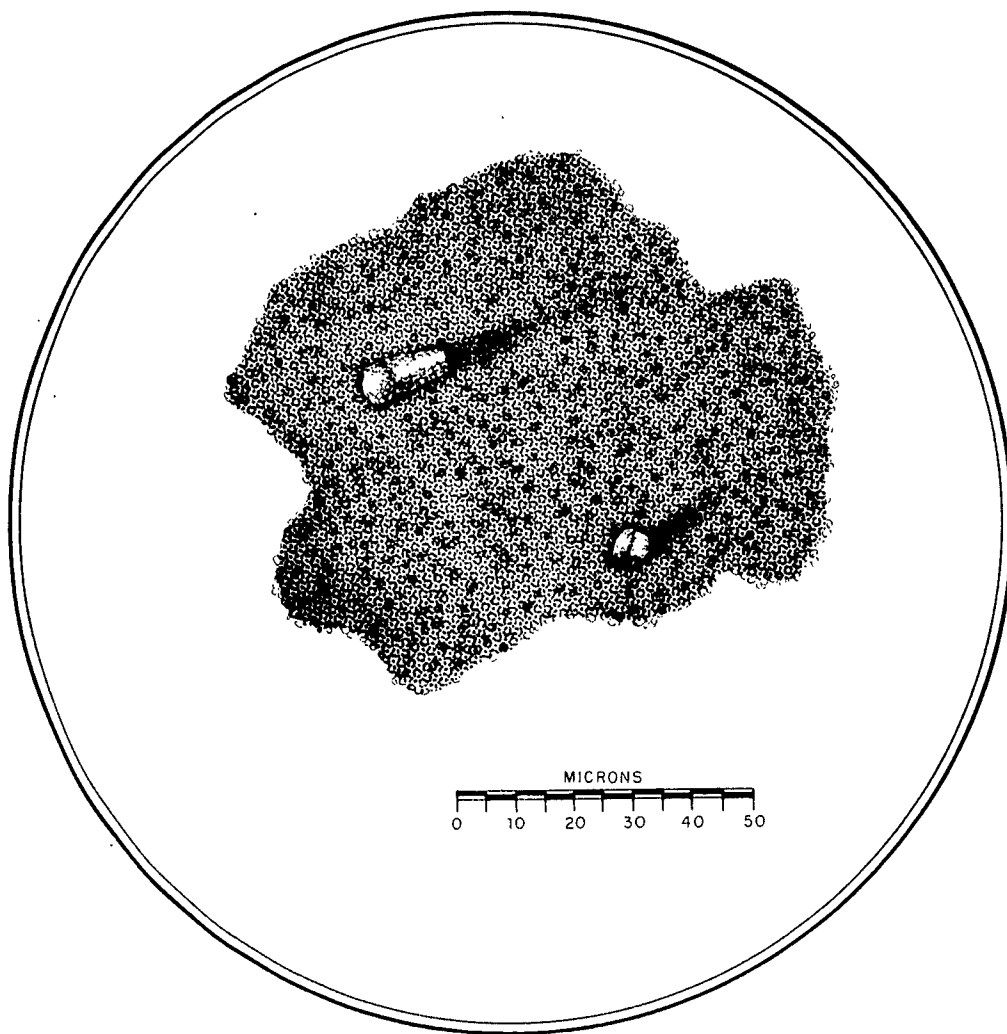


Fig. 21 Artist's drawing of microscopic (X500) appearance of gold black.

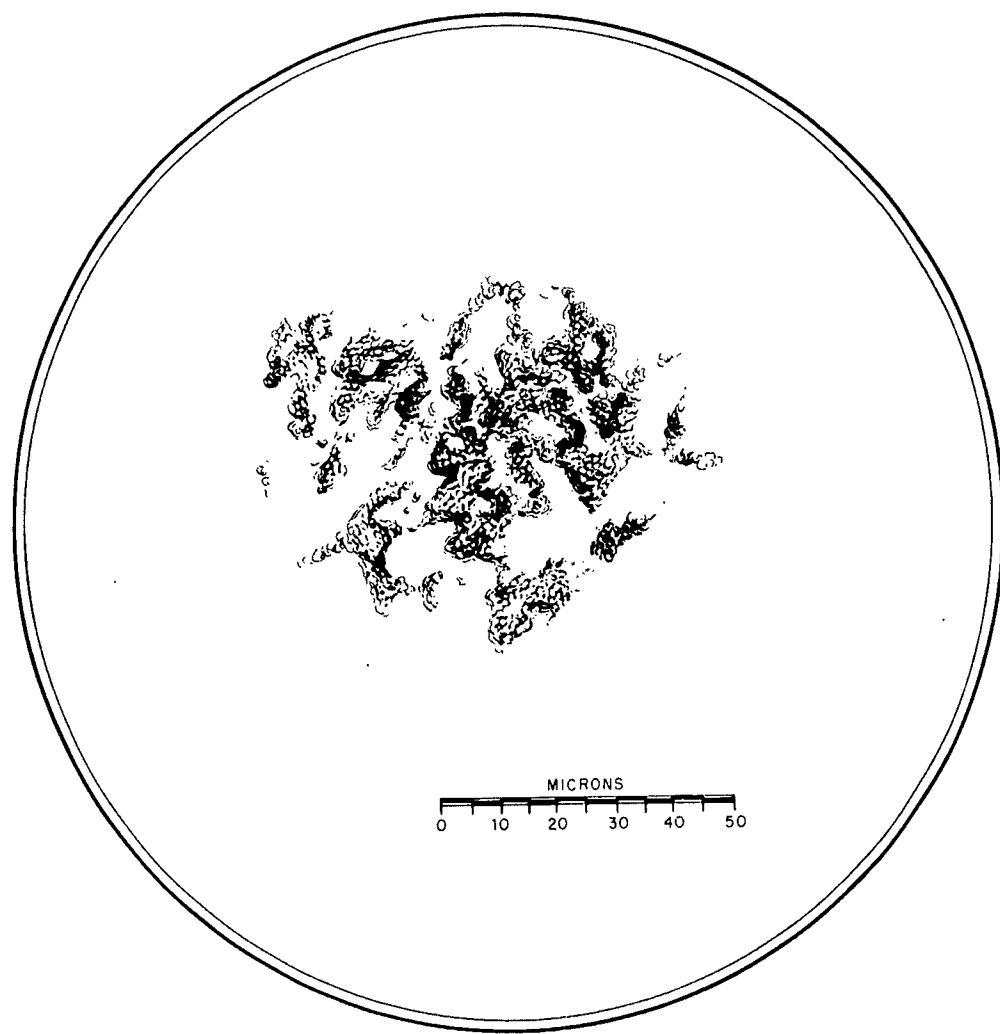


Fig. 22 Artist's drawing of microscopic (X500) appearance of Parson's Black Optical Lacquer.

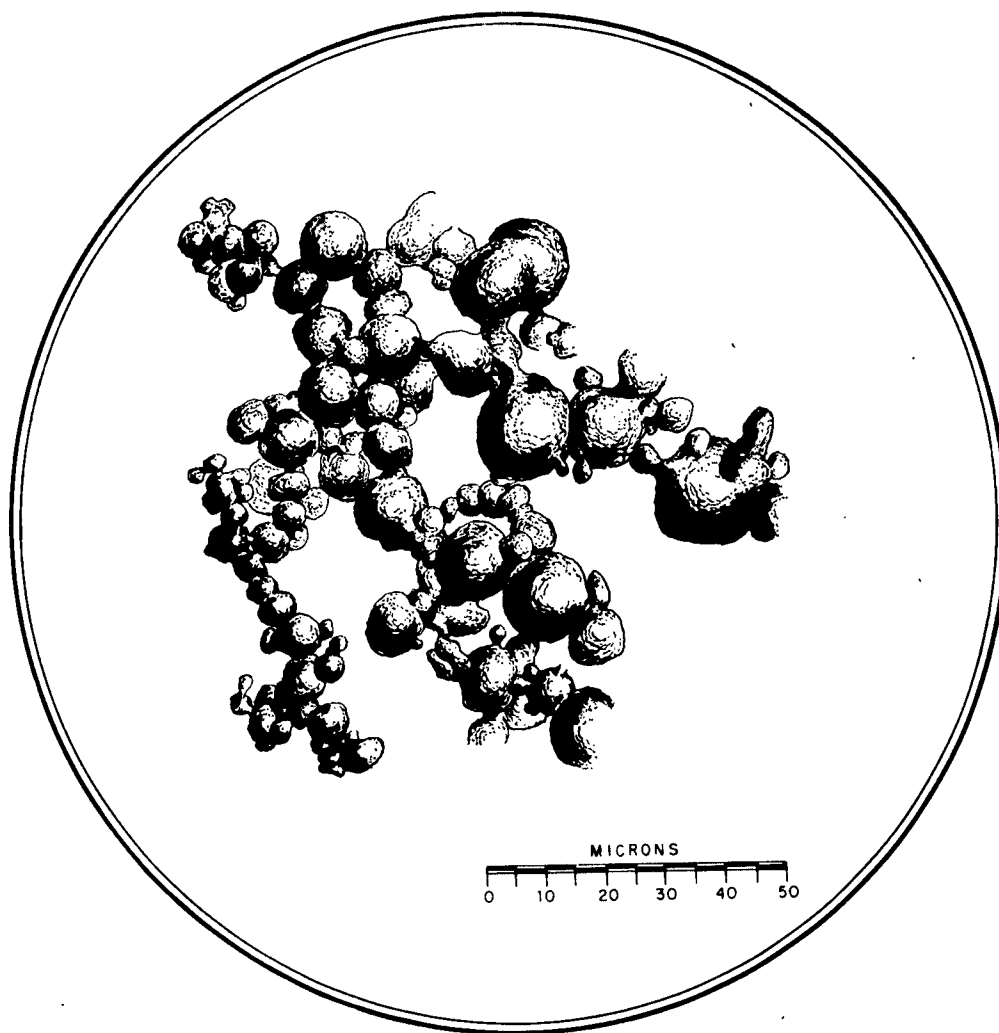


Fig. 23 Artist's drawing of microscopic (X500) appearance of 3M velvet black.

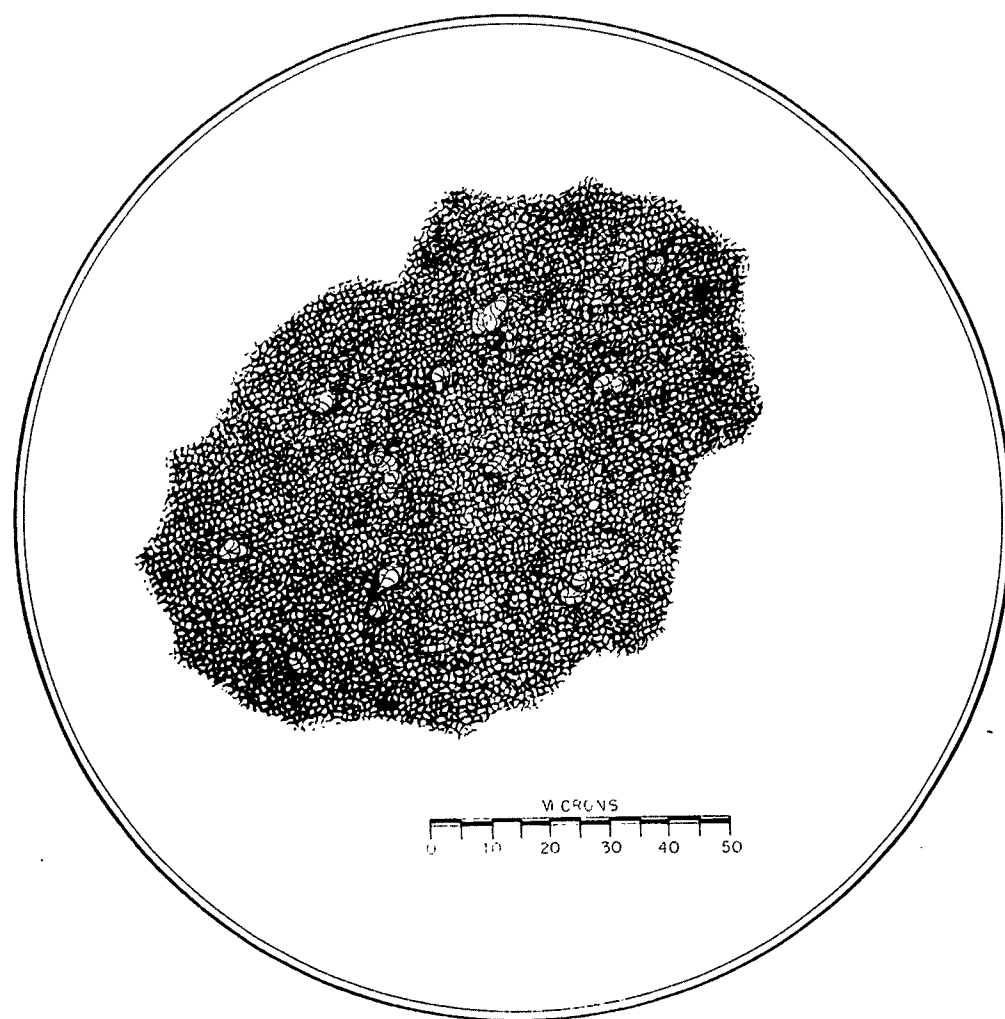


Fig. 24 Artist's drawing of microscopic (X500) appearance of Fuller metal etching primer.

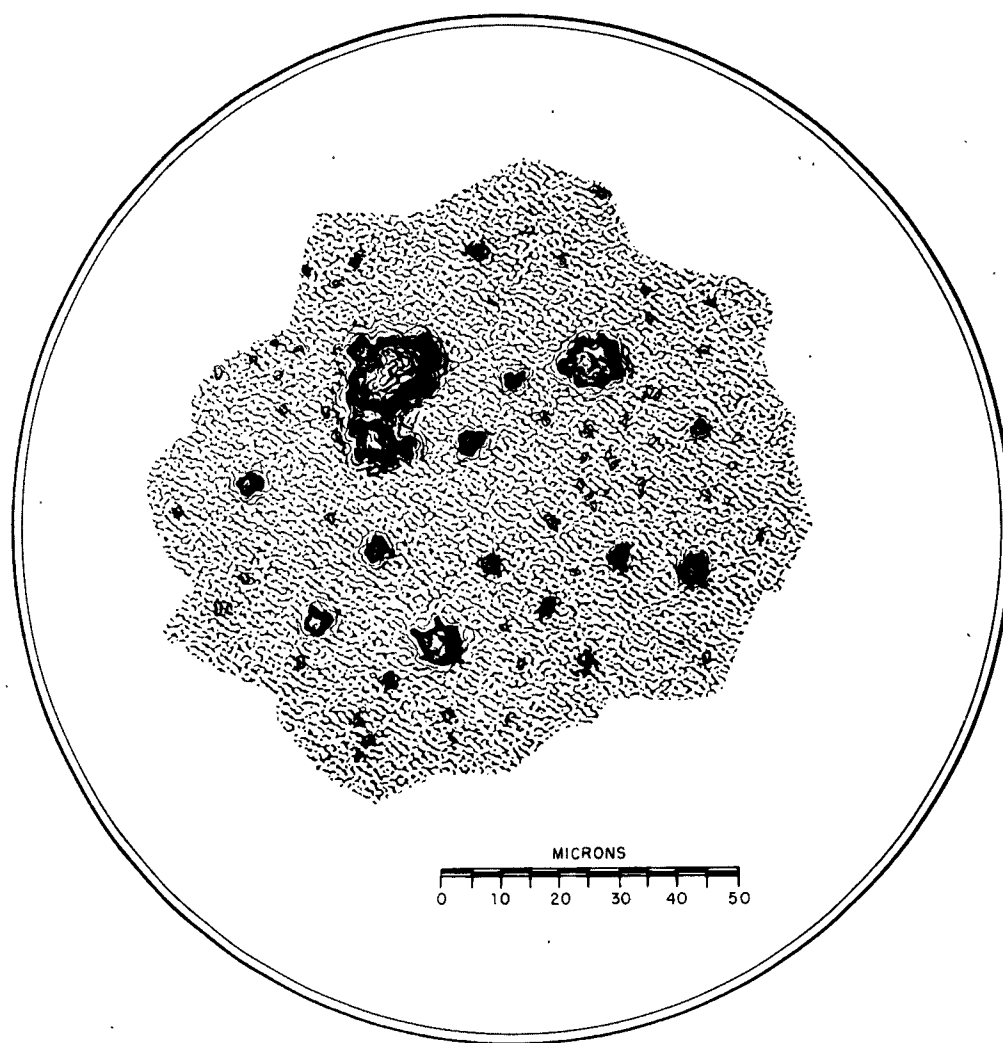


Fig. 25 Artist's drawing of microscopic (X500) appearance of Fuller velvet black plastic enamel.

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<p>Analytical and experimental analysis show that errors inherent in the method are less than 1% of the emittance of a black body at the temperature of the measurement, although instrumental errors and uncertainties in the heat capacities of the specimens reduce the accuracy of the reported data to $\pm 10\%$. The blackest material measured was electrically deposited platinum black on gold. The emittance of a black paint appeared to be more dependent on the thickness that it could be applied than on the specific type of paint. Heavily anodized aluminum exhibited an emittance near 77°K of about 0.6, nearly as high as the best black paints, although it looked bright to the eye. The best platinum black coating had an emittance above 0.9 at 77°K. The metal blacks and the black paints had emittances above 0.95 at room temperature.</p> <p>This technical documentary report has been reviewed and is approved.</p> <p>UNCLASSIFIED</p>	<p>Analytical and experimental analysis show that errors inherent in the method are less than 1% of the emittance of a black body at the temperature of the measurement, although instrumental errors and uncertainties in the heat capacities of the specimens reduce the accuracy of the reported data to $\pm 10\%$. The blackest material measured was electrically deposited platinum black on gold. The emittance of a black paint appeared to be more dependent on the thickness that it could be applied than on the specific type of paint. Heavily anodized aluminum exhibited an emittance near 77°K of about 0.6, nearly as high as the best black paints, although it looked bright to the eye. The best platinum black coating had an emittance above 0.9 at 77°K. The metal blacks and the black paints had emittances above 0.95 at room temperature.</p> <p>This technical documentary report has been reviewed and is approved.</p> <p>UNCLASSIFIED</p>